

991,500

AMENDED SPECIFICATION

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PATENT SPECIFICATION

DRAWINGS ATTACHED

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Int. Cl.:—C 07 d // B 04 d

COMPLETE SPECIFICATION

Process and apparatus for the Purification of Glucose  
Syrup and Dextrose Juice

We, WESTFALIA SEPARATOR AKTIEN-  
GESELLSCHAFT, a German Company, of  
Werner-Habig-Strasse, Oelde, Westfalen,  
Germany, do hereby declare the invention, for  
5 which we pray that a patent may be granted  
to us, and the method by which it is to be  
performed, to be particularly described in and  
by the following statement:—

The invention relates to a process and  
10 apparatus for the preliminary purification of  
glucose syrup and dextrose juice, by means  
of which associated constituents, largely  
proteins and fats, are separated out. In the  
following description of the invention, starch  
15 will be considered as the starting material in  
the production of the glucose syrup and  
dextrose juice.

The extraction of starch from cereals and  
20 chopped fruits, e.g. sorghum, tapioca, batatas  
(sweet potatoes), maize, wheat, rice and  
potatoes, is a well known process. However,  
the starch obtained from these raw materials  
is commercially available as such, in part only;  
25 following extraction, a considerable proportion  
of the starch is converted to saccharified starch  
products for which there are a multitude of  
uses in industry.

The saccharification of the starch can be  
effected by means of an acidic hydrolytic pro-  
30 cess or by enzymatic means, or, again, by a  
combination of these two processes. In the  
interests of simplicity, in the following descrip-  
tion the acidic hydrolytic process only will  
be described in detail.

35 Acidic hydrolysis is effected by the conver-  
sion of an acidified starch milk, under pressure.  
The hydrogen ions in the acid effect a catalytic  
splitting of the starch such that the starch  
molecules are broken down to form the end-  
40 product of hydrolysis, i.e., grape sugar of  
[Price 4s. 6d.]

composition  $C_6H_{12}O_6$ . The grape sugar can  
take the form either of a glucose syrup, in  
which case it can have the most varied com-  
positions, or of a pure dextrose juice depend-  
ing upon the way in which the process is  
effected.

In the case of incomplete breakdown, besides  
pure dextrose the end-product contains higher  
molecular breakdown products such as maltose  
and oligosaccharides. The presence of these  
oligosaccharides prevents the grape sugar from  
crystallising out. An incompletely broken down  
product of this sort is generally termed a  
glucose or starch syrup.

With complete or near-complete saccharifi-  
cation of the starch, the proportion of oligo-  
saccharides is so small that the grape sugar  
can crystallise out of the solution. Such solu-  
tions are termed dextrose juices since it is  
from these that the grape sugar, generally  
referred to as dextrose, can be extracted in  
pure crystalline form.

During the subsequent neutralisation of the  
acid, which has been employed purely as a  
catalyst, using for instance soda or calcium  
carbonate, the precipitable proteins are flo-  
culated. At the same time, water-soluble  
sodium chloride or calcium chloride is formed,  
if hydrochloric acid was used as the catalyst,  
this being accompanied by the development of  
carbon dioxide.

Instead of using acidic hydrolysis, the break-  
down of the starch to form glucose syrup or  
dextrose juice can also be effected by employ-  
ing specific enzymes and maintaining certain  
breakdown conditions; it is likewise possible,  
as previously mentioned, to combine acidic  
hydrolysis with enzymatic breakdown.

Since, in addition to starch, the starting  
material also contains small amounts of non-

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- saccharific substances, such as albumen and fats, these, to a large extent, reappear unmodified in the glucose syrup or dextrose juice.
- 5 According to the present invention, a process for the purification of glucose syrup or dextrose juice includes subjecting the syrup or juice at a pH of between 3.5 and 6.0 to a preliminary purification to form at least two fractions in a centrifugal separator having a bowl adapted for rotation about a vertical axis and provided with a plurality of spaced frustoconical plates, arranged between an inner collecting chamber for a lighter fraction containing a substantial portion of the proteins and fats contained as solid impurities in the starting material and an outer collecting chamber for at least one heavier fraction comprising the bulk of the liquid phase, delivering the juice to be centrifuged into the inner chamber which constitutes the collecting chamber for the lighter fraction and from which the heavier fraction flows outwards through the spaces between the frusto-conical plates and separately removing the lighter fraction from the inner chamber and the heavier fraction from the outer chamber. Preferably the heavier fraction comprising the bulk of the liquid is fed by means of a paring element to a stationary outlet which may conveniently be equipped with a throttling device to enable the position of the separation zone between the two fractions within the bowl to be altered.
- For separating liquid/solid mixtures, in which the solids have a lower specific weight than the carrier liquid, it is not usual to employ centrifugal separators, since the removal of these solids from the central part of the separator bowl is accompanied by considerable difficulties. For this reason, other separating devices are usually preferred, in all cases where mixtures of this sort are involved. It has been discovered, however, that with glucose syrups and dextrose juices, at the pH value range specified the solids occur in a form which makes them amenable to removal if the direction of flow through the plates is outwards.
- By employing a centrifugal separator to effect a preliminary purification of the solutions in question, up to 96% of the solids they contain can be separated from the liquid phase. The process can be operated on a practically continuous basis since the flow factor for a centrifugal separator is generally constant while with a plate filter this factor reduces considerably as blockage increases. Owing to the fact that by far the majority of the solids and fats are separated from the liquid phase in the separator, the activity of the filter carbon is maintained through a substantially longer period. If a centrifugal separator is employed in the manner set forth to effect a preliminary purification of the solution in question, the considerable amount of manual labour necessary to keep the filters operative is saved. Besides this, there is a considerable reduction in the consumption of filter media and the effectiveness of the active carbon is maintained for a considerably longer interval so that the requirement in this respect is essentially reduced.
- In general, the solutions to be centrifuged greater specific weight than the carrier liquid in the form of insoluble components. In order to prevent these from being deposited upon the inner wall of the bowl and thus gradually building up in the direction towards the bowl axis, it is recommended that a type of bowl be employed which ejects the heavier solids, together with part of the liquid, in the form of a second heavier fraction, through openings provided in its periphery. To this end, a nozzle-type separator can be used, this being provided with permanently open nozzles distributed uniformly around the bowl periphery. The mixture of heavier solids plus liquid, issuing from these nozzles, can then rejoin the juice from which the proteins and fats have been extracted, and be further processed together with this.
- However, to the same end, a separator having an automatically opening bowl can be employed from which the solids are ejected, in concentrated form and at longer intervals, together with a small quantity of liquid. In this case, the solid concentrate can be taken out of the process altogether.
- The final purification of solutions pre-purified in accordance with the present invention, is a matter which depends upon the judgement and experience of the operative concerned. As a general rule, however, further filtration in a carbon filter is carried out, the operational life of such filter amounting to many times that obtainable with the conventional processes owing to the excellent preliminary purification. It may also be left to the expert operative's judgement to use, after the separator, any filter which the installation contains. Since the pre-purified solutions only contain a small residual percentage of solids, the operational life of a plate filter of this sort is very appreciably extended.
- Since, in glucose syrups, the proteins flocculate in coarser fashion than with dextrose juices, when the first-mentioned material is being centrifuged, these solids, together with a proportion of the juice, must be floated out of the central collecting chamber. It is therefore necessary to follow the separator with a rotary sieve or the like, in order once again to separate this liquid from the solids. This liquid can then rejoin the main body of the juice and the whole be further processed.
- Where dextrose juices are being processed, a rotary sieve or similar device is unnecessary.
- The invention will now be explained in more detail with reference to the accompanying drawings in which:—
- Figure 1 shows a flow sheet for the process-

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ing of glucose syrup according to the invention;

Figure 2 shows a similar flow sheet for the processing of dextrose juice;

5 Figure 3 is a side view partly in section of one half of a separator bowl for the continuous extraction of proteins and fats using paring discs; and

10 Figure 4 shows the upper portion of a modified form of the bowl shown in Figure 3.

Referring to Figure 1, the starch milk, diluted with water, is fed through a pipe 1 to a container 2 which is provided with a stirrer 3 in order to prevent precipitation of the starch grains. Where the acidic hydrolytic process is employed, the starch milk is passed from the container 2 through a pipe 4 to a converter 5 and there has added to it a small quantity, e.g. 0.2%, of hydrochloric acid which is fed in through the pipe 6. The converter 5 can be either of the continuous or non-continuous type. The starch, broken down to a greater or lesser degree, passes through a pipe 7 to a neutralising vessel 8 to which a neutralising agent such as soda or calcium carbonate is fed through a pipe 9. The vessel 8 is also provided with a stirrer 10. The reaction mixture is then fed through a pipe 11 to a centrifugal separator 12 from which the lighter fraction containing the proteins and fats leaves through a pipe 13, and, the juice from which most of the solids are removed leaves through a pipe 14. If a nozzle-type separator is employed, the mixture of fluid and heavier solids, ejected through the nozzles on the periphery of the bowl, is fed through a pipe 15 to join the juice leaving through the pipe 14 and the mixture thus formed is further processed. In order to prevent blocking of the nozzles, the inlet passage is provided with duplicate sieves 16 which are operated alternately, one being in operation whilst the other is being cleaned. Where a separator having an automatically desludging bowl is used, the ejected solids of higher specific weight are taken out of the process via the pipe 15.

During the centrifugal processing of glucose syrup, the lighter solids floated out of the bowl together with a portion of the liquid, are fed through the pipe 13 to a rotary sieve 17 or the like, which once again separates the liquid from the solids. This liquid is conveniently passed back through a pipe 18 to the separator inlet 11, the solids being tapped off through a pipe 19.

The flow sheet illustrated in Figure 2 relates to the purification of dextrose juices. It is distinguished from the flow sheet of Figure 60 1 only by the fact that the rotary sieve 17 and the recirculation pipe 18 are omitted.

The separator bowl for use in the process of the present invention illustrated in Figure 3 by way of example, comprises a lower section 20 and an upper section 21 which are held

together by means of a threaded ring 22. The material to be processed is passed through a supply tube 23 into the inlet space 24 of the bowl and flows from there through drillings 25 in a distributor 26, into the central section 27 of the separating space which also constitutes an inner collecting chamber. The heavier liquid and the heavier solids migrate outwardly through the spaces between a plurality of frusto-conical plates 28 and force the lighter proteins and fats inwards. These latter after surmounting a weir 29 pass into a paring chamber 30, where they are skimmed off by a stationary paring disc 31 and discharged through a skimming passage 32.

70 The main body of the juice, proteins and fats removed, flows through passages 33 between the upper section 21 of the bowl and a separating disc 34, into a second paring chamber 35 where it is picked up by a paring disc 36 and discharged through a passage 37. In the discharge pipe (not shown) connected to the passage 37, a throttle valve is situated in order, by varying the back pressure, to shift the position of the separating zone indicated at 38.

75 The heavier solids issue, together with a portion of the juice, through nozzles 39 at the bowl periphery. This mixture joins the juice discharged through the passage 37 and the mixture thus formed is fed onwards for further processing.

80 In the modified bowl shown in Figure 4, the solids comprising the proteins and fats and passing into the chamber 30 via the weir 29, are flung into a receptacle (not shown) through small tubes 40, whilst the sugar juice, as with the bowl of Figure 3, is extracted by means of a paring disc 36.

#### 90 WHAT WE CLAIM IS:—

100 1. A process for the purification of glucose syrup or dextrose juice which includes subjecting the syrup or juice at a pH of between 3.5 and 6.0 to a preliminary purification to form at least two fractions in a centrifugal separator having a bowl adapted for rotation about a vertical axis and provided with a plurality of spaced frusto-conical plates, arranged between an inner collecting chamber for lighter fraction containing a substantial portion of the proteins and fats contained as solid impurities in the starting material and an outer collecting chamber for at least one heavier fraction comprising the bulk of the liquid phase, delivering the juice to be centrifuged into the inner chamber which constitutes the collecting chamber for the lighter fraction and from which the heavier fraction flows outwards through the spaces between the frusto-conical plates and separately removing the lighter fraction from the inner chamber and the heavier fraction from the outer chamber.

110 2. A process according to Claim 1, wherein the heavier fraction containing the bulk of the

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- liquid phase is fed to a stationary outlet by means of a paring element.
3. A process according to Claim 2, wherein said stationary outlet is provided with a throttling device whereby the position of the separation zone within the separator may be altered.
4. A process according to any of claims 1 to 3, wherein solids of higher specific gravity than the heavier fraction are separately removed from the separator together with a portion of the liquid as a second heavier fraction.
5. A process according to Claim 4, wherein said second heavier fraction is recombined with the first heavier fraction after both said fractions have been removed from the separator.
6. A process according to Claim 4 or Claim 5, wherein said second heavier fraction is removed through nozzles situated at the periphery of the separator bowl.
7. A process according to Claim 4 or Claim 5, wherein said second heavier fraction is removed periodically through openings provided in the peripheral wall of the separator bowl.
8. A process according to Claim 7 wherein the separator bowl is provided with a plurality of normally closed apertures in the peripheral wall thereof, said apertures being adapted to be periodically opened to permit the discharge of the second heavier fraction.
9. A process according to any of the preceding Claims, as applied to the purification of glucose syrup wherein the lighter fraction removed from the separator contains an appreciable proportion of liquid, and wherein after said lighter fraction leaves the separator, the liquid contained therein is separated and recycled to the glucose syrup fed to the separator.
10. A process according to Claim 9, wherein said liquid is separated from said lighter fraction in a rotary sieve.
11. A process as claimed in anyone of the preceding claims wherein said inner collecting chamber is defined by the inner ends of the frusto-conical plates and the outer wall of a distributor surrounding an axially disposed inlet for the glucose syrup or dextrose juice, said distributor being provided with apertures for the passage of liquid to be centrifuged from said inlet to said inner collecting chamber.
12. A process according to claim 11, wherein said outer collecting chamber is defined by the outer ends of said plates and the inner peripheral wall of said bowl.
13. A process according to any one of the preceding claims wherein means for removing the heavier liquid fraction from the outer collecting chamber comprise a paring chamber communicating with said outer collecting chamber, and a stationary paring disc adapted to feed said heavier fraction from said paring chamber into a stationary discharge pipe.
14. A process according to any of the preceding claims wherein means for removing the lighter fraction from said inner collecting chamber comprising a second paring chamber mounted above said inner collecting chamber and communicating therewith over a weir, and a second stationary paring disc adapted to feed said lighter fraction from said second paring chamber into a second discharge pipe.
15. A process for the purification of glucose syrup or dextrose juice substantially as hereinbefore described with reference to Figure 1 or Figure 2 of the accompanying drawings.
16. A process for the purification of glucose syrup or dextrose juice substantially as hereinbefore described and using apparatus as described with reference to Figures 3 and 4 of the accompanying drawings.

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329 High Holborn.

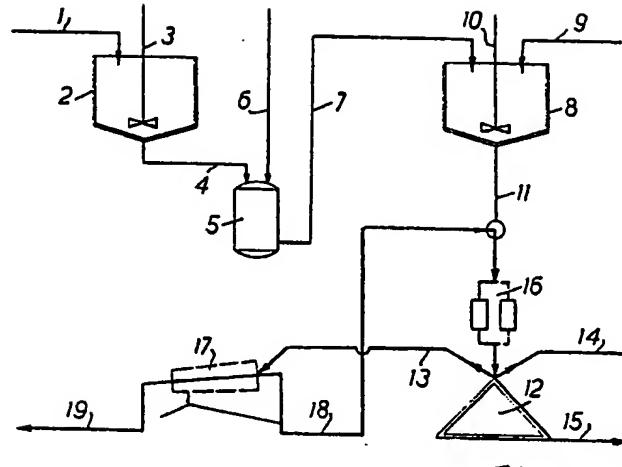


FIG. 1.

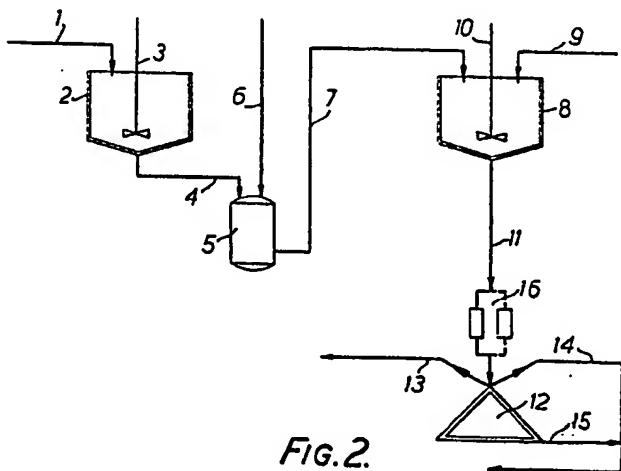


FIG. 2.

991500 AMENDED SPECIFICATION  
2 SHEETS *This drawing is a reproduction of  
the Original on a reduced scale  
Sheets 1 & 2*

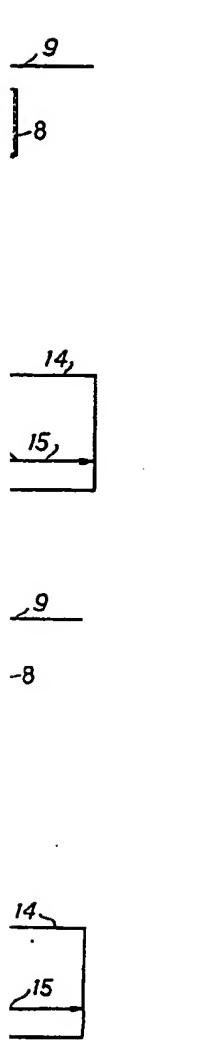


FIG.4.

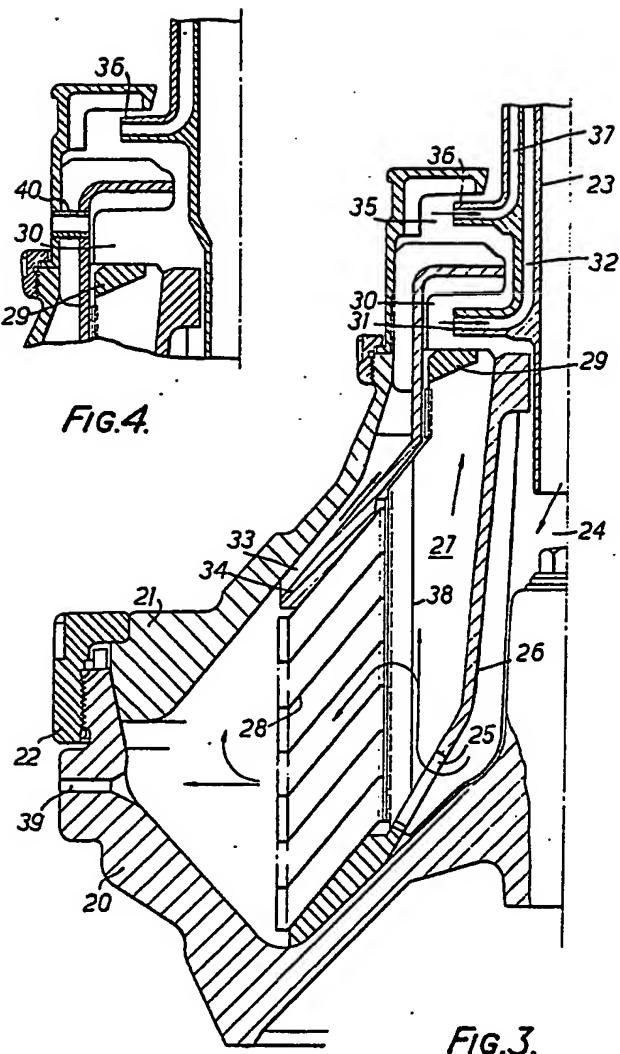


FIG.3.

991500 AMENDED SPECIFICATION  
2 SHEETS This drawing is a reproduction of  
the Original on a reduced scale  
Sheets 1 & 2

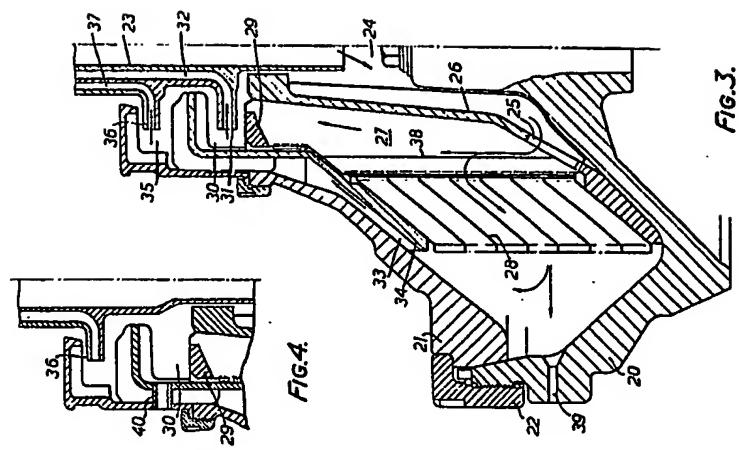


FIG.3.

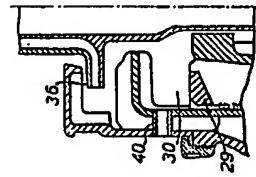


FIG.4.

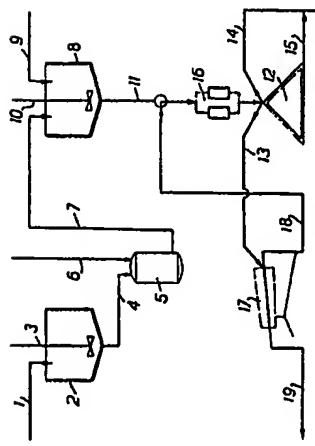


FIG.1.

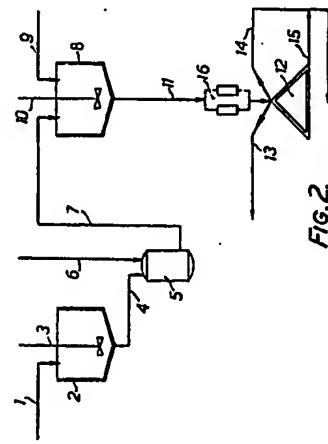


FIG.2.